

09/341921

510 Rec'd PCT/PTO 21 JUL 1999

TRANSMITTAL LETTER TO THE UNITED STATES

ATTORNEY'S DOCKET NUMBER 47699

DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)

INTERNATIONAL APPLICATION NO.
PCT/EP 98/00480INTERNATIONAL FILING DATE
29 January 1998PRIORITY DATE CLAIMED
31 January 1997

TITLE OF INVENTION: PURIFICATION OF ETHYLENE OXIDE BY DISTILLATION

APPLICANT(S) FOR DO/EO/US Bernd BESSLING, Hans HASSE, Juergen PLUECKHAN, Thomas MAYER, Heinz AUER

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. /X/ This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.
 2. / / This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.
 3. /X/ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
 4. /x / A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
 5. /X/ A copy of the International Application as filed (35 U.S.C. 371(c)(2)).
 - a./X/ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b./ / has been transmitted by the International Bureau.
 - c./ / is not required, as the application was filed in the United States Receiving Office (RO/USO).
 6. /X/ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
 7. / / Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)).
 - a./ / are transmitted herewith (required only if not transmitted by the International Bureau).
 - b./ / have been transmitted by the International Bureau.
 - c./ / have not been made; however, the time limit for making such amendments has NOT expired.
 - d./ / have not been made and will not be made.
 8. / / A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
 9. /X/ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
 10. / / A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).
- Items 11. to 16. below concern other document(s) or information included:
11. / / An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
 12. /X/ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
 13. /X/ A FIRST preliminary amendment.
/ / A SECOND or SUBSEQUENT preliminary amendment.
 14. / / A substitute specification.
 15. / / A change of power of attorney and/or address letter.
 16. /X/ Other items or information.
International Search Report
International Preliminary Examination Report

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U.S. Appln. No. (If Known)

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PCT/EP 98/00480ATTORNEY'S DOCKET NO.
47699

	CALCULATIONS	PTO USE ONLY
17. /X/ The following fees are submitted		
BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)):		
Search Report has been prepared by the		
EPO or JPO.....	\$840.00	840.00
International preliminary examination fee paid to USPTO		
(37 CFR 1.482).....	\$750.00	
No international preliminary examination fee paid to		
USPTO (37 CFR 1.482) but international search fee paid		
to USPTO (37 CFR 1.445(a)(2)).....	\$700.00	
Neither international preliminary examination fee		
(37 CFR 1.482) nor international search fee		
(37 CFR 1.445(a)(2)) paid to USPTO	\$ 970.00	
International preliminary examination fee paid to		
USPTO (37 CFR 1.482) and all claims satisfied pro		
-visions of PCT Article 33(2)-(4).....	\$96.00	

ENTER APPROPRIATE BASIC FEE AMOUNT = \$ 840.00

Surcharge of \$130.00 for furnishing the oath or declaration
later than / / 20 / / 30 months from the earliest
claimed priority date (37 CFR 1.492(e)).

Claims	Number Filed	Number Extra	Rate
Total Claims	10	-20	X\$18.
Indep. Claims	2	-3	X\$78.
Multiple dependent claim(s) (if applicable)			+270.
TOTAL OF ABOVE CALCULATION			= 840.00

Reduction of 1/2 for filing by small entity, if applicable.
Verified Small Entity statement must also be filed
(Note 37 CFR 1.9, 1.27, 1.28).

SUBTOTAL = 840.00

Processing fee of \$130. for furnishing the English
translation later than / / 20 / / 30 months from the
earliest claimed priority date (37 CFR 1.492(f)). +

TOTAL NATIONAL FEE = 840.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)).
The assignment must be accompanied by an appropriate cover
sheet (37 CFR 3.28, 3.31) \$40.00 per property =

TOTAL FEES ENCLOSED = \$ 880.00

Amount to be
refunded: \$
Charged \$

a./X/ A check in the amount of \$ 880. to cover the above fees is enclosed.

b./ / Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.

c./X/ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 11-0345. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

KEIL & WEINKAUF
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SIGNATURE

Herbert B. Keil
Herbert B. Keil
NAME
18,967
Registration No.

09/341921
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of)
BESSLING et al.)
) BOX PCT
International Application)
PCT/EP 98/00480)
)
Filed: January 29, 1998)
)
For: PURIFICATION OF ETHYLENE OXIDE BY DISTILLATION

PRELIMINARY AMENDMENT

Honorable Commissioner of
Patents and Trademarks
Washington, D.C. 20231

Sir:

Prior to examination, kindly amend the above-identified application as follows.

IN THE CLAIMS

Claim 4, line 1, delete "or 3".

Claim 6, line 1, delete "one of the claims 1 to 5" and insert --claim 1--.

Claim 7, line 1, delete "one of the claims 1 to 6" and insert --claim 1--.

Claim 8, line 1, delete "one of the claims 1 to 7" and insert --claim 1--.

Claim 9, line 1, delete "one of the claims 1 to 8" and insert --claim 1--.

Claim 10, lines 1 and 2, delete "one of the claims 7 or 9" and insert --claim 7--.

R E M A R K S

The claims have been amended to eliminate multiple dependency and to put the case in better form for U.S. filing. No new matter is included.

Favorable action is solicited.

Respectfully submitted,

KEIL & WEINKAUF

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As originally filed

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Purification of ethylene oxide by distillation

10

15

The invention relates to processes and an apparatus for the purification of ethylene oxide by distillation.

Pure ethylene oxide is a product manufactured worldwide in amounts of several million tonnes per year. The last process step in the preparation of pure ethylene oxide is purification by distillation, with ethylene oxide being isolated from an aqueous solution.

The critical factor in the purification of ethylene oxide by distillation is that aldehydes, in particular formaldehyde and acetaldehyde, which are present in the feed, do not pass into the pure product. The most important reasons for formaldehyde having to be separated off in the purification of ethylene oxide by distillation are high product purity requirements (frequently below 10 ppm of total aldehyde) and the introduction of novel catalysts which lead to higher concentrations of formaldehyde in the feed.

GB-B 1,180,822 discloses a process for separating off formaldehyde from an ethylene-oxide-containing aqueous mixture. The degree of purity of the pure ethylene oxide achieved in the distillation substantially depends in this process on the amount of fresh water used as scrubbing water. To achieve low formaldehyde contents in the pure oxide, high scrubbing water flow rates must be used, which increases the waste water load. The process is therefore inexpedient.

EP-B 0 322 323 discloses a process for separating off aldehyde impurities from crude ethylene oxide by distillation, in which the crude ethylene oxide is introduced into a column, which has 50 theoretical plates, at the height of the 30th plate from the top. The ethylene oxide is obtained as top product with a content of
5 from about 0.0015 to 0.0020% by weight of aldehyde impurities. The liquid stream exiting at the bottom of the column contains the water present in the crude ethylene oxide and ethylene oxide at 0.15 to 3 times the amount by weight of water - the bottom phase is thus not ethylene-oxide-free, which is a disadvantage in processing terms, since purification by distillation in this case can only be
10 operated economically in conjunction with a glycol plant. The high ethylene oxide content in the bottom phase, in addition, has the consequence that there are only small temperature differences between top and bottom in the column used for purification of ethylene oxide by distillation in the known process.

15 Because of the disadvantages in the abovementioned processes for purification of ethylene oxide by distillation, process are customarily presently used in which the pure ethylene oxide is obtained as a side stream: US 4,134,797 discloses, for example, a process for purification of ethylene oxide by distillation, in which crude ethylene oxide, which is contaminated with aldehydes such as formaldehyde
20 and acetaldehyde, is purified in a column by means of fractionation or via a plurality of gas-liquid contact stages. The crude ethylene oxide is introduced into the column at a height of preferably from 1 to 20 theoretical gas-liquid contact stages. The pure oxide having a content of generally less than 20 ppm of formaldehyde is obtained as a side stream. The top product obtained is an
25 ethylene-oxide-containing, formaldehyde-enriched stream. The disadvantages of this process are the high expenditure on equipment, the increase in the amount of pure ethylene oxide, which causes safety problems, in the column and the fact that crude oxide is only partially converted into pure oxide (contaminated top product).

It is an object of the present invention, therefore, to provide a process which is simple to perform and at the same time enables a product to be produced, in the purification of ethylene oxide by distillation, which is substantially free of formaldehyde. For the purposes of the invention, substantially free means, for example, that starting from a content of approximately 50 ppm or more in the feed the purified ethylene oxide obtained contains only approximately 4 ppm or less formaldehyde. Furthermore, the process shall also satisfy stringent safety requirements. In addition, it shall lead to an ethylene oxide which is free from formaldehyde to a high degree, without scrubbing steps involving high amounts of waste water.

We have found that this object is achieved according to the invention by a process for the purification of ethylene oxide by distillation, comprising the step in which an aqueous mixture comprising ethylene oxide, formaldehyde and at least 5% by weight of water is introduced via a feed into a distillation apparatus comprising at least one distillation column, the mixture being introduced at a height above the bottom of at least 8, preferably from 12 to 56, theoretical stages, the ethylene oxide is taken off at the top and in the bottom phase a mixture is obtained which contains less than 5% by weight, preferably less than 0.05% by weight, of ethylene oxide.

In addition, to carry out the process according to the invention, an apparatus is provided which comprises a distillation column having a feed (1) at a height above the bottom (4) of at least 8 theoretical stages or, in the case of a plate column, at least 12 plates above the bottom (4), a top take-off (3), optionally a side take-off (5), and also flame-arresting packings and optionally an intermediate reboiler between feed (1) and bottom (4).

In another embodiment, a process is provided for purification of ethylene oxide by distillation, which process comprises the step in which an aqueous mixture

comprising ethylene oxide, formaldehyde and at least 5% by weight of water is introduced via a feed into a distillation apparatus comprising at least one packed column which contains a structured or bulk packing and has a specific mass transfer area A, the mixture being introduced at a height above the bottom of at least x_{\min} (in m) which, for a given specific mass transfer area A (in m^2/m^3), is given by the equation

$$x_{\min} = 5.5 \text{ m} - A \bullet 0.006 \text{ m}^2,$$

the ethylene oxide is taken off at the top and in the bottom phase a mixture is obtained which contains less than 5% by weight, preferably less than 0.05% by weight, of ethylene oxide.

In a further embodiment, a process for the purification of ethylene oxide by distillation is provided, which comprises the step in which an aqueous mixture comprising ethylene oxide, formaldehyde and at least 5% by weight of water is introduced via a feed into a distillation apparatus comprising at least one plate column, the mixture being introduced at a height above the bottom of at least 12, preferably from 16 to 84 plates, the ethylene oxide is taken off at the top and in the bottom phase a mixture is obtained which contains less than 5% by weight, preferably less than 0.05% by weight, of ethylene oxide.

The crude ethylene oxide fed comprises ethylene oxide, formaldehyde and at least 5% by weight, preferably from 20 to 60% by weight, of water.

25

In all said embodiments, it is particularly preferred if the process is conducted in such a manner that the bottom mixture contains less than 100 ppm of ethylene oxide. ppm, here and elsewhere, are by weight. At such low concentrations of ethylene oxide in the bottom phase of the column - highly predominantly aqueous bottom product - the bottom temperature in the column is far higher than the

temperature at the top of the column where there is pure ethylene oxide. In the column, there is then, above the bottom, a spatially narrow region in which the temperature changes sharply.

- 5 A feature of the process according to the invention is that the feed into the purification by distillation is markedly above the temperature jump over the bottom in the column. The crude ethylene oxide is introduced according to the invention at a height of at least 8, preferably from 12 to 56, theoretical stages, or at at least 12, preferably from 18 to 84, plates over the bottom.

10

If a packed column, containing structured or bulk packings, is used for the separation, the crude ethylene oxide is introduced at a minimum height x_{\min} given by the abovementioned equation as a function of the specific mass transfer area; preferably, the aqueous mixture is introduced via the feed at a height of from 1.5
15 x_{\min} to 7 x_{\min} . For example, for a specific mass transfer area of $250 \text{ m}^2/\text{m}^3$, the equation gives a minimum height of 4 m, preferably an introduction height of from 6 to 28 m. In the case of a specific mass transfer area of $500 \text{ m}^2/\text{m}^3$, this gives a minimum introduction height of 2.5 m, a preferred introduction height of from 3.75 m to 17.5 m.

20

In the process according to the invention, the crude ethylene oxide can also be introduced via a plurality of feed lines. The lowest of the plurality of feed lines is preferably situated in this case at the abovementioned distance from the bottom according to the invention. The process can also be carried out in such a manner
25 that more than one column is used. The minimum distance according to the invention is then preferably maintained between the crude ethylene oxide feed point or points and the bottom.

In the influent aqueous mixture, the formaldehyde is predominantly present in the
30 form of methylene glycol, which is formed in an equilibrium reaction with water.

By introducing according to the invention the aqueous mixture into the distillation apparatus at a certain, as defined above, (minimum) distance from the bottom, the zone between the feed and the bottom of the column becomes according to the invention so long that monomeric formaldehyde, which is released in the bottom
5 phase as a result of the high temperatures, owing to the equilibrium lying on the side of monomeric formaldehyde at high temperatures, is reabsorbed by the feed stream running in countercurrent.

In addition, in a particularly preferred embodiment of the process according to the
10 invention, it is provided that residues of formaldehyde which, despite the above-described measure, pass into the enrichment part of the column, are scrubbed out by feeding a small stream of water into the enrichment part of the column; ie., that at a height of at least 1 theoretical stage or plate, preferably 3 to 20 theoretical stages or plates, above the feed of the aqueous mixture comprising ethylene oxide,
15 formaldehyde and said minimum amount of water, a further mixture, principally comprising water or essentially water alone, is additionally introduced via a feed line. Feed values of about 0.02 for the ratio mass of water/mass of ascending gas stream are sufficient here. The monomeric formaldehyde released in the bottom phase is preferably absorbed while still in the stripping part of the purification by
20 distillation by the abovementioned further aqueous feed stream. Residues of monomeric formaldehyde which pass into the enrichment part are thus scrubbed out by the added water.

In the accompanying drawing,

25

Fig. 1 shows a preferred embodiment of the apparatus used to carry out the process according to the invention.

The figure shows an embodiment in which the process is implemented in a single
30 column. Between the feed (1) point of the crude ethylene oxide, which comprises

water, acetaldehyde and formaldehyde, and the bottom phase (4) of water, acetaldehyde and formaldehyde, the minimum distance according to the invention, as explained above, is maintained. In the preferred embodiment according to Fig. 1, the further aqueous mixture, preferably water, which serves to absorb
5 residues of monomeric formaldehyde, is fed at the point marked by (2). The product - pure ethylene oxide - is taken off overhead (3).

In the process according to the invention, a substantially formaldehyde-free product is taken off at the top of the column. Between the feed (1) and the bottom
10 take-off (4), in a particularly preferred embodiment there is a side take-off (5) via which a stream of ethylene oxide, water, acetaldehyde and formaldehyde is taken off which is enriched with acetaldehyde in comparison with the feed. To reduce operating costs, in a further particularly preferred embodiment of the process according to the invention between the feed (1) and the bottom take-off (4) there is
15 provided an intermediate reboiler into which heat is introduced at a lower temperature than in the bottoms reboiler. In addition, in a very particularly preferred embodiment of the process according to the invention, for safety reasons, flame-arresting arranged packings, as are described, for example, in WO 97/19069, are used in the column or at the feeds and/or take-offs to achieve the
20 separation action. Typical values for the concentration of formaldehyde in the product are below from 1 to 2 ppm.

Example

25 The process according to the invention was investigated by a works test in an ethylene oxide plant [site: Ludwigshafen]. In this test, at formaldehyde concentrations in the feed of about 170 ppm, a formaldehyde concentration in the top product of 2 ppm was achieved.

We claim:

5

1. A process for purification of ethylene oxide by distillation, comprising the step in which

10 - an aqueous mixture comprising ethylene oxide, formaldehyde and at least 5% by weight of water is introduced via a feed into a distillation apparatus comprising at least one distillation column, the mixture being introduced at a height above the bottom of at least 8 theoretical stages,

15 - the ethylene oxide is taken off at the top and

15

 - in the bottom phase, a mixture is obtained which contains less than 5% by weight of ethylene oxide.

- 20 2. A process for purification of ethylene oxide by distillation, comprising the step in which

25 - an aqueous mixture comprising ethylene oxide, formaldehyde and at least 5% by weight of water is introduced via a feed into a distillation apparatus comprising at least one packed column which contains a structured or bulk packing and has a specific mass transfer area A , the mixture being introduced at a height above the bottom of at least x_{\min} (in m) which, for a given specific mass transfer area A (in m^2/m^3), is given by the equation

$$x_{\min} = 5.5 \text{ m} - A \cdot 0.006 \text{ m}^2,$$

30

- the ethylene oxide is taken off at the top and

- in the bottom phase a mixture is obtained which contains less than 5% by weight of ethylene oxide.

5

3. A process as claimed in claim 2, wherein the aqueous mixture is introduced via the feed at a height of from $1.5 x_{\min}$ to $7 x_{\min}$.

10

4. A process as claimed in claim 2 or 3, wherein the specific mass transfer area A is in the range from $100 \text{ m}^2/\text{m}^3$ to $500 \text{ m}^2/\text{m}^3$.

15

5. A process as claimed in claim 1, wherein the aqueous mixture comprising ethylene oxide, formaldehyde and at least 5% by weight of water is introduced via a feed into a distillation apparatus comprising at least one plate column, the mixture being introduced at a height above the bottom of at least 12 plates.

20

6. A process as claimed in one of the claims 1 to 5 which further comprises the step in which a further mixture, principally comprising water, is additionally introduced via a feed line at a height of at least one theoretical stage or plate above the feed of the aqueous mixture.

25

7. A process as claimed in one of the claims 1 to 6, wherein flame-arresting packings are used in the distillation apparatus.

30

8. A process as claimed in one of the claims 1 to 7, wherein a distillation apparatus is used in which, between the feed and the bottom, there is installed a side take-off via which is taken off a mixture which is enriched with acetaldehyde in comparison with the influent aqueous mixture.

9. A process as claimed in one of the claims 1 to 8, wherein a distillation apparatus is used in which an intermediate reboiler is situated between the feed and the bottom.
- 5 10. An apparatus for carrying out the process as claimed in one of the claims 7 or 9, comprising a distillation column having a feed (1) at a height above the bottom (4) of at least 8 theoretical stages or, in a plate column, of at least 12 plates above the bottom, a top take-off (3), optionally a side take-off (5), and flame-arresting packings and, optionally, an intermediate reboiler between
10 feed (1) and bottom (4).

Abstract

5

In a process for purification of ethylene oxide by distillation an aqueous mixture comprising ethylene oxide, formaldehyde and at least 5% by weight of water is introduced via a feed into a distillation apparatus comprising at least one distillation column, the mixture being introduced at a height above the bottom of at
10 least 8 theoretical stages, the ethylene oxide is taken off at the top and in the bottom phase a mixture is obtained which contains less than 5% by weight of ethylene oxide.

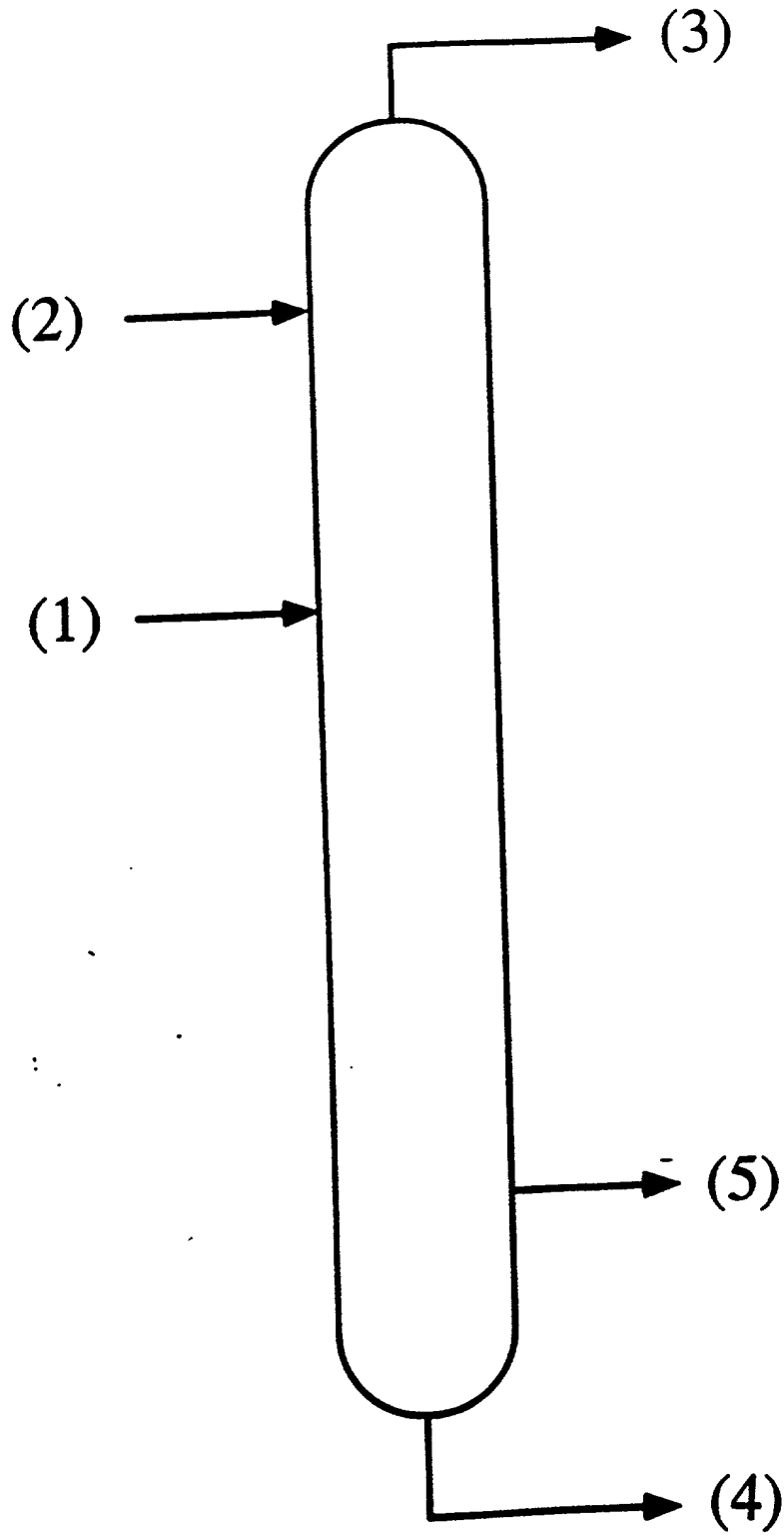


Fig. 1

Declaration, Power of Attorney

Page 1 of 4

O. Z. 0050/47699

We (I), the undersigned inventor(s), hereby declare(s) that:

My residence, post office address and citizenship are as stated below next to my name,

We (I) believe that we are (I am) the original, first, and joint (sole) inventor(s) of the subject matter which is claimed and for which a patent is sought on the invention entitled

Purification of ethylene oxide by distillation

the specification of which

☒ is attached hereto.

☐ was filed on _____ as

Application Serial No. _____

and amended on _____.

☒ was filed as PCT international application

Number PCT/EP 98/00480

on 29 January 1998

and was amended under PCT Article 19

on _____ (if applicable).

We (I) hereby state that we (I) have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

We (I) acknowledge the duty to disclose information known to be material to the patentability of this application as defined in Section 1.56 of Title 37 Code of Federal Regulations.

We (I) hereby claim foreign priority benefits under 35 U.S.C. § 119(a)-(d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed. Prior Foreign Application(s)

Application No.	Country	Day/Month/Year	Priority Claimed
19703627.9	Federal Republic of Germany	31st January 1997	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No

O. Z. 0050/47699

We (I) hereby claim the benefit under Title 35, United States Codes, § 119(e) of any United States provisional application(s) listed below.

 (Application Number)

 (Filing Date)

 (Application Number)

 (Filing Date)

We (I) hereby claim the benefit under 35 U.S.C. § 120 of any United States application(s), or § 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application.

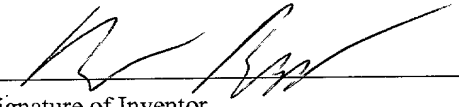
Application Serial No.	Filing Date	Status (pending, patented, abandoned)
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And we (I) hereby appoint **Messrs. HERBERT. B. KEIL**, Registration Number 18,967; and **RUSSEL E. WEINKAUF**, Registration Number 18,495; the address of both being Messrs. Keil & Weinkauff, 1101 Connecticut Ave., N.W., Washington, D.C. 20036 (telephone 202-659-0100), our attorneys, with full power of substitution and revocation, to prosecute this application, to make alterations and amendments therein, to sign the drawings, to receive the patent, and to transact all business in the Patent Office connected therewith.

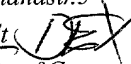
We (I) declare that all statements made herein of our (my) own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

O. Z. 0050/47699

Bernd Beßling
NAME OF FIRST OR SOLE INVENTOR



Signature of Inventor

Date May 7, 1999


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Jürgen Plückhan
NAME OF THIRD JOINT INVENTOR

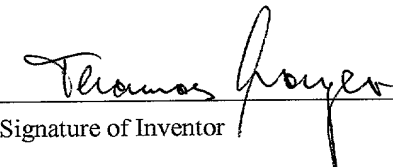

Signature of Inventor

Date May 7, 1999

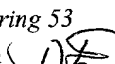
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Thomas Mayer
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Signature of Inventor

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Citizen of: Germany
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Declaration

Page 4 of 4

O. Z. 0050/47699

50 Heinz Auer

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Signature of Inventor

Citizen of: Germany

Post Office Address: same as residence

Date May 7, 1999

68809 Neußheim